

UPGRADING OF TACONITE CONCENTRATE
TO DIRECT REDUCTION SPECIFICATIONS BY
FLOTATION

by

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and

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ABSTRACT

This paper describes the results of tests to float silicates in sea water from Sydvaranger taconite concentrate. The concentrate grade had to be raised from 67% to approximately 70.5% Fe. The superconcentrate could then be used as feed for a direct reduction process.

Silicates present were quartz, cummingtonite, and hornblende. Quartz was easy to remove by cationic flotation. Most of the report deals therefore with the flotation of the two other gangue minerals.

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A/S Sydvaranger has mined taconite for 68 years in Northern Norway close to the Russian border.⁽¹⁾ The ore body contains 30% magnetite and traces of hematite. The major gangue mineral is quartz, but relatively large amounts of amphiboles can occur in part of the ore body. The amphibole ore zones are troublesome because the amphiboles are even more interlocked with the magnetite than is the quartz, and the amphiboles are also more resistant to fine grinding than the other minerals.

Concentration is now done by coarse crushing, magnetic cobbing, fine crushing, coarse grinding-magnetic separation, and fine grinding to 75-81% minus 325 mesh and magnetic separation. Sea water is used throughout the process**, because fresh water is in short supply.

The concentrate is pelletized in Allis Chalmers grate kilns. Production is 2.5 million metric tons of pellets annually, containing 65% Fe.

The company got an early option on Norwegian North Sea Royalty gas for further processing of its pellets. A Midrex reduction plant is now being built in Emden, North Germany as a 50-50% venture with the German firm Korf Stahl. The pellets currently produced contain too much silica for direct reduction feed. Silica can be removed in three ways. One is finer grinding and subsequent magnetic separation. This would be expensive and result in a concentrate too fine for pelletization. Another alternative is fine screening. Table 1 shows the weight and iron distribution in screen fractions of a typical concentrate. Total analysis is here 67% Fe, and analysis of the -74 micron size is 68.8% Fe. Screening was piloted

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**Except in the last magnet separation stage.

in the plant, but the tests were not successful because of marine growth and corrosion of the screens, and because tough coarse amphibole-rich middlings built up in the grinding circuit.

The third alternative, which we will deal with here, is silicate flotation. Early flotation test work was done prior to the installation of the pellet plant in the 1960's to obtain a better pellet grade. It was then demonstrated that pellet-quality concentrate could be obtained by flotation. This was done in a sea-water pulp by cationic collectors in a basic circuit, with guar gum or dextrine as iron depressant. Flotation of particles coarser than 147 microns was difficult. The process was similar to many processes developed at that time except for the utilization of sea water. However, simultaneous improvement in magnetic separator design made flotation unnecessary.

When work to meet D-R requirement started, we could build on the former experience. Specification for final concentrate grade could not be firmly established, but market research indicated that the price of metallized pellet would depend highly upon purity. Our aim for concentrate grade through the test work was 70.5% Fe (tot), which corresponds to about 1.5% SiO₂.

TABLE 1

WEIGHT AND IRON DISTRIBUTION ON SIZE FRACTIONS
OF A MAGNETIC SEPARATOR CONCENTRATE (FLOTATION FEED)

<u>Fraction</u> <u>Micron</u>	<u>% Wt</u>	<u>% Iron</u>	
		<u>Analysis</u>	<u>Distribution</u>
+147	1.5	35.5	0.8
-147+74	8.3	55.7	6.9
-74+43	24.2	66.7	24.1
-43	66.0	69.5	68.2
Total	100.0	67.0	100.0

FLOTATION FEED

Moist samples of magnetic separator concentrate were received from the plant. Samples received at different times varied in mineral composition and iron grade, dependent on which pit was being mined. The flotation characteristics of the sample would deteriorate a little during storage, and the natural pH would tend to drop. Almost all gangue in the magnetic concentrate is present as locked particles or gangue with small inclusions of magnetite.

Two methods of dissolving Fe for chemical analyses were used. One, designated Fe(HCl), does not dissolve much iron in silicates such as amphibole. The other, designated Fe(tot), will dissolve iron in silicates. An empirical formula was developed for the amount of amphibole in a given sample.

$$\% \text{ Amph} = 5 (\text{Fe}(\text{tot}) - \text{Fe}(\text{HCl}))$$

The use of this formula together with microscopic examinations showed that the composition of the magnetic separator concentrates was in the following range: magnetite 90-92%, quartz 4-7%, amphiboles 3-5%. The magnetic separator concentrate will, later in the paper, be called mag-product not to confuse it with the flotation concentrate.

FLOTATION CONSTRAINTS

The supply of fresh water available to the concentrator is limited; therefore, it is preferable to utilize as much sea water as possible in the flotation. The sea water content can be brought down to 30% without having to dewater the mag-product. The concentration of Mg^{++} and Ca^{++} will be in the magnitude of 10^{-2} M, which will render the use of anionic collectors most difficult.

Maintaining satisfactory grade with all feed types requires either 100% removal of quartz or some amphibole removal. The flotation of amphibole is therefore stressed throughout this work.

Sydvaranger is situated at 70° northern latitude. Pulp temperatures will be in the range 5-12° C during the year. The effects of this temperature range upon flotation had to be investigated.

Jacobs et al (2) have reported that the commonly used iron depressants, guar gum and dextrine at high pH, cause severe filter problems. One gets poor filtration rates as well as blockage of the filter cloth. Minimization of depressant addition is therefore desirable.

BATCH TEST WORK

Initial tests on mag-product were run to confirm earlier results. The original flowsheet, with use of Armac C as a collector, guar gum as depressant at pH 10.2, and with lime as pH modifier in fresh water, worked well on a sample with 1.2% amphibole and only 1.5% +147 micron oversize. On a sample with 3% amphibole and 2.7% +147 micron, the results were much poorer, as shown in Table 2. The use of 100% sea water deteriorated the flotation still further.

The amphibole content and fineness of grind, which are

interrelated, are clearly more important than flotation water composition. Subsequent pilot plant tests demonstrated that the amphibole problem was even more important in continuous operation.

TABLE 2

BATCH TEST RESULTS WITH ARMAC C AS A COLLECTOR,
SHOWING THE INFLUENCE OF AMPHIBOLE CONTENT IN
THE FEED AND PROCESS WATER TYPE

<u>% Amphibole</u>	<u>Water</u>	<u>% Fe(HCl) in Conc.</u>	<u>% Fe Recovery</u>
1.2	fresh	70.4	97.7
3.0	fresh	70.1	94.3
3.0	sea	69.8	95.2

When the role played by the amphibole minerals was established, a designated test series was run to gather insight into the flotation of amphiboles with cationics in sea water. A special sample high in amphibole was handpicked in the mine. After magnetic separation and desliming, the composition of this sample was: 55% magnetite, 25% amphiboles and 20% quartz. The test work on amphiboles was primarily carried out with products from Azko Chemie because this company provides a relatively complete series of amines and systematic product information. Variables were amine chain lengths and number of amine groups. Flotation conditions and results, as percent of each mineral floated, are listed in Table 3.

The collector dosage was unfortunately chosen a little too low so total recoveries are not impressive. It is clearly demonstrated, anyway, that diamines are more effective and more selective than primary amines, and that longer chains are more effective than shorter chains. The quaternary amine EN 24 was effective, but the amount required was excessive. In the last two tests with the best reagent, pH was increased, and in the very last, depressant was added. Those tests are not comparable to the others because of much higher reagent additions. It can be seen that the depressant depresses both magnetite and amphibole.

The tests on the amphibole ore then provided basis for a similar test series on mag-products. A larger number of commercial collectors were tried, mainly on a diamine basis. The tests were run at natural pH, without depressant addition, at 10° C, in 30% sea water. On the basis of test results, two sets of ranking lists could be set up.

<u>Highest Fe Grade at 95% Rec.</u>	<u>Highest Amt. of Amphibole Removal</u>
Duomac T	Aerosurf MG 83A
Duomac C	Duomac T
Aerosurf MG 83A	Duomac C
Aerosurf MG 70 A 3	-
Aerosurf MG 98 A 3	-
Lilafлот D 810	-

Because amphibole removal was the principal aim of this part of the investigation, Aerosurf MG 83A was chosen for further test work.

A small test series was run with various iron depressants; and Orfloc 59, a dextrine type, was chosen for additional study.

TABLE 3

RECOVERY OF MINERALS FROM AMPHIBOLE-RICH SAMPLE AT pH 8, IN
40% SEA WATER, AT 20° C

Collector Dosage = 25 + 25 + X g/t
Frother Flotanol D 16 20 g/t

<u>Reagent</u>	<u>Dosage(g/t)</u>	<u>% of Each Mineral Floated</u>		
		<u>Amphibole</u>	<u>Quartz</u>	<u>Magnetite</u>
Duomac T	50	21	48	10
Duomac C	50	19	36	10
Armac T	50	19	23	11
Armac C	50	16	19	10
EN 24	350	40	82	26
Duomac T*	175	61	86	34
Duomac T**	175	50	87	30

* pH 9, Frother MIBC

** pH 9, Guar gum 420 g/t, Frother MIBC

Two important variables in flotation were amount of depressant, which we wanted to minimize, and pH. Table 4 shows the results from tests with those two variables. The results are expressed in three parameters: maximum concentrate grade, corresponding amphibole removal and iron loss. The amphibole figures are calculated from the difference between two large numbers and might be a little erratic.

The tests do not lead to a sharply defined optimum condition, but conclusions can be drawn. High pH's give good iron depression, but amphibole is depressed as well. The result is relatively low grades. The same can be said about the highest depressant additions. The low pH of 5.5 gives surprisingly good iron recoveries and a fair amphibole float but not the very best grades. No depressant addition, which could be preferable from a filtration point of view, results in poorer recoveries.

pH 8, the natural pH, is easiest to maintain. Results here are good, especially when 50-200 g/t of depressant is added, both when grade-recovery and amphibole float are considered.

Most of the batch and pilot work was carried out at the prevailing air and water temperatures. Special tests had to be run to find the effect of the plant water temperature. In Table 5 data from two test series are shown, one with depressant addition and high collector consumption, and one without depressant and with low collector. It can be seen that grade at a given recovery is about 5% poorer at the low pulp temperature. Amphibole and magnetite are easier to float at the lower temperature while the quartz float appears to be poorer.

PILOT FLOTATION TEST WORK

Pilot tests were run to verify batch results and to spot problem areas that required further batch work. The pilot plant in Trondheim is relatively small and can handle from 100 to 300 kilos per hour. A fair choice of Denver and Agitair type cells is available.

The moist mag-product received from the mine was slurried in a 1-cu-meter agitated tank and fed as a slurry to the flotation circuit via an automatic valve, controlled by an electromagnetic flow meter. A flowsheet which includes upgrading of all middlings is shown in Figure 1. It consists of pH adjustment, mixing of depressant and collector, and direct float of gangue. The non-float is finished concentrate. The float fraction is cleaned by flotation, regrinding and magnetic separation to increase the magnetite recovery. In many tests, the middlings treatment was somewhat simplified. A special feature of the flowsheet is that no middlings are circulated back to the head

TABLE 4

FLOTATION RESULTS GIVEN AT DIFFERENT PH'S AND DEPRESSANT ADDITIONS

30% Sea Water. Collector: Aerosurf MG 83A 30 + 20 + 20 g/t
 MIBC 35 g/t Depressant: Orfloc 59 pH Regulators: H2SO4 - NaOH
 Temp 9-10° C

pH	Depressant Addition (g/t)				
	0	50	100	200	400
Grade	69.2	70.2	69.7	69.6	-
10.0	52	21	36	33	-
Amph.	8	9	9	4	-
Loss					
Grade	70.4	71.3	70.8	70.4	70.8
9.0	53	23	70	53	0
Amph.	9	11	12	8	6
Loss					
Grade	71.3	71.3	71.2	71.1	70.9
8.0	87	58	86	70	70
Amph.	22	17	17	13	11
Loss					
Grade	71.3	71.2	71.5	71.3	71.6
7.0	63	100	75	86	0
Amph.	27	29	28	20	14
Loss					
Grade	70.2	70.8	70.6	70.6	70.5
5.5	85	57	58	71	85
Amph.	15	16	16	14	13
Loss					

Grade = Fe_(tot) content of concentrate in %
 Amph. = % of amphibole in feed floated
 Loss = % of iron in feed floated
 Feed grade Fe(HCl) : 66.3%
 Fe_(tot) : 66.8%

TABLE 5

EFFECT OF TEMPERATURE UPON FLOTATION
AT NATURAL pH, IN 30% SEA WATER

Depressant Orfloc 59(g/t)	200		0	
Collector Aerosurf MG 83A(g/t)	70		35	
Frother MIBC (g/t)	35		17	
	<u>23° C</u>	<u>10° C</u>	<u>18° C</u>	<u>10° C</u>
% Fe(HCl) in conc at 95% rec.	69.9	69.5	70.1	69.3
% Quartz floated*	93.0	84.0	58.0	68.0
% Amphibole floated*	50.0	70.0	63.0	69.0
% Magnetite floated*	10.0	13.0	5.0	9.0
Feed % Fe(HCl)		66.6		

*% mineral floated means total amount floated at the end of the test.

end of the rougher flotation. The reason is that desliming is unnecessary before this flotation because most fine gangue particles are removed by magnetic separation. The ones left float well and do not cause any problems as long as they are prevented from circulating. Return of any middlings resulted in the buildup of a heavy circulating load and prevented flotation of coarse quartz.

For practical reasons, only 10% sea water was used in our pilot tests. Major variables to be examined during the pilot tests were: collector - primary amine at high pH versus diamine at natural pH, feed composition, and the effect of depressants.

TABLE 6

PILOT PLANT RESULTS WHEN ARMAC C IS USED AS COLLECTOR,
MIBC AS FROTHER AND GUAR GUM(450 g/t)AS DEPRESSANT
IN 10% SEA WATER, AT pH 9.8-10.2

	Test 31 <u>Amphibole-Rich Feed</u>	Test 203 <u>Quartz-Rich Feed</u>
Feed % Fe(HCl)	67.2	67.6
Conc % Fe(HCl)	70.1	71.1
Rec. % Fe	95.0	95.0
Total Grade % Fe(HCl)	-	71.1
Total Rec. % Fe(HCl)	-	97.0

Note:

Conc. grade and rec. take only the direct float into consideration, while total grade and recovery include upgraded middlings.

Table 6 shows results on two ores when Armac C is used as a collector at pH 10.2-9.8. The amphibole-rich feed gives a concentrate with 1% lower grade than that with quartz-rich feed. Recoveries are generally good and middlings treatment is not essential.

Table 7 shows results with Aerosurf MG 83A as collector. A good grade can be obtained with both quartz- and amphibole-rich feeds but at the expense of recovery. Magnetite recovered from middlings will have a grade lower than 68% Fe in the amphibole case and will dilute the primary concentrate.

Use of depressants appears not to be essential when there is much amphibole in the feed.

In Table 8 some screen analyses of products from test 19, which was not successful, are given. The table demonstrates in which screen fractions there are problems.

TABLE 7

PILOT PLANT RESULTS WHEN AEROSURF MG 83A IS USED AS COLLECTOR, MIBC AS FROTHER AND ORFLOC 59 AS DEPRESSANT - 10% SEA WATER

	Test 202 <u>High Quartz</u>	Test 205 <u>High Amphibole</u>	Test 208 <u>High Amphibole</u>
Depressant	175 g/t	175 g/t	0
pH	nat. 8	nat. 7.8	raised to 8
Feed % Fe _(HCl)	67.2	66.4	66.6
Conc % Fe _(HCl)	71.1	71.1	70.7
Rec. % Fe _(HCl)	87	73	85
Final Conc % Fe _(HCl)	71.0	70.2	70.3
Final Rec. % Fe _(HCl)	91	93	95

Note: Conc. grade and rec. take only direct float into consideration, while the recovery from middlings is included in the final values. Fe values are based on HCl dissolution.

Not much silicate is floated in the coarse size range, the +147 micron fraction of the final concentrate contains only 39.9% Fe and the -147+74 micron fraction 58.7%. This is not much improvement over the flotation feed. Eighty percent of the total weight of the concentrate is in the -44 micron fraction which contains 70% Fe. The reason that the overall grade is not more than 68.4% Fe is the failure to float coarse silicates.

The screen analyses of the first and second float products show that the major iron losses are in the -20 micron fraction, where iron generally should be liberated. The fine particles are probably trapped in the froth which is fairly stiff. Return of middlings to the rougher circuit will, as mentioned before, bring those fines into circulation.

TABLE 8

SCREEN ANALYSES OF PRODUCTS FROM PILOT PLANT TEST 19 WITH
 AN AMPHIBOLE-RICH FLOTATION FEED
 ARMAC C USED AS COLLECTOR AT PH 10.2 REGULATED WITH LIME.
 DEPRESSANT: GUAR GUM 400 g/t

Fraction Microns	Feed		Iron Concentrate			First Float			Second Float		
	Wt %	Fe %	Wt %	Fe %	Fe Distr %	Wt %	Fe %	Fe Distr %	Wt %	Fe %	Fe Distr %
+147	2.1	38.8	1.9	39.9	1.1	2.8	10.4	1.0	4.0	15.4	1.6
-147+ 74	6.4	50.4	5.7	58.7	4.9	11.5	11.6	4.7	13.3	17.1	5.9
-74+ 44	11.6	60.7	11.3	66.2	10.9	14.7	13.1	6.8	14.7	21.6	8.2
-44+ 20	39.4	69.0	41.2	70.0	42.2	25.2	16.0	14.2	21.0	39.3	21.4
-20	40.5		39.9	70.0	40.9	45.8	45.4	73.3	47.0	51.5	62.9
Calc. Fe %		66.2		68.4			28.1			39.3	
Analyzed Fe%		66.0		68.9			27.7			39.4	
Wt Distr.		100.0		91.0			1.5			7.5	
Fe Distr.		100.0		94.9			0.6			4.5	

Note: % Fe = Fe(HCl)

AMINE DISTRIBUTION IN PULP

Because a disposal permit is required for any industrial effluent to air or water in Norway, an investigation of the only reagent thought harmful-amine- was undertaken. A good analytical procedure has been described by Bahr(3). The amine level was not only determined in the final tailings but throughout the whole flotation circuit.

Measurements were done during pilot test 206 where amphibole-rich mag-product was floated with Aerosurf MG 83A at pH 8.2. Water samples for analysis were pipetted out from the pulp samples after settling.

Amine additions and pulp water concentrations are given in mg/l in Figure 2. It was observed that adsorption was rapid and that most of the amine added actually is adsorbed on the minerals or in the froth phase. Pumping of the pulp causes desorption, while increase of surface during regrinding gives some adsorption. Only 6% of the added amine ended in the tailing water phase and 4% in the concentrate water phase. The flotation tailings will be mixed with the magnetic separator tailings in the plant, and the surplus amine should be adsorbed on the large silicate surfaces present there.

ZETA POTENTIAL MEASUREMENT

The literature gives some information on the zeta potentials of the minerals concerned. The method of measurement is not consistent through different works, nor has the effect of sea water been investigated. Because a Riddick Zeta Meter (4) was available at the Minerals Research Laboratory in Asheville, it was decided to carry out some determinations. The zeta potential was calculated by use of the formula:

$$S = 113000 \frac{\eta}{\epsilon} \frac{V}{E}$$

where: ϵ = Dielectric constant

E = Electric field strength

V = Particle velocity

η = Viscosity of water

Calculations were done graphically by a chart supplied with the Zeta Meter. No corrections for the influence of ionic strength of the solution were made. Mineral samples from the mine were too fine grained for separation prior to measurements.

Samples from other places had therefore to be used with the differences in composition that might result. Particulars about the minerals are given in Table 9.

TABLE 9
MINERALS USED FOR ZETA POTENTIAL MEASUREMENTS

Location	<u>Quartz</u>	<u>Magnetite</u>	<u>Cumingtonite</u>	<u>Hornblende</u>
	Spruce Pine, N. C.	Ontario	North Dakota	Ontario
% Fe	0.002	68.5	23	18
% Al ₂ O ₃	0.006	-	8.8	8.8
% SiO ₂	-	-	44.3	48.6
% CaO	0.006	-	1.3	9.6

A 0.5 g -20+100 mesh sample was wet ground in an agate mortar under conductivity or sea water to approximately 30% -10 micron. The coarse was removed by settling. The minerals were at no stage of their treatment in contact with any acid, base or salt not used for the actual measurement. Samples ground in conductivity water were stored in 500 ml water for 30 minutes plus the time necessary for measurement of previous samples. Each measurement took approximately 5 minutes after mixing with sulfuric acid or sodium hydroxide solution. Both storage in conductivity water and storage at a given pH could influence the subsequently measured zeta potential as demonstrated by Ney⁽⁵⁾. The retention times chosen were as near as possible to retention times encountered in flotation. Samples ground in sea water were stored for one hour in 4% and measured in 2% sea water. This concentration is much lower than the 30% to be used in the plant, but measurements are difficult and inaccurate at high ionic strengths.

Figure 3 shows the results in conductivity water. Zero point of charge for quartz, 2.0, corresponds well to the value quoted by Fuerstenau and Palmer⁽⁶⁾, 1.8, while the magnetite value, 4.8, is lower than the 6.5 quoted by them but closer to the value given by Ney⁽⁵⁾, 4.0. The curves indicate that quartz should be easy to separate from the other minerals at all pH's with cationic collectors. The curves for magnetite, cumingtonite and hornblende are quite close and demonstrate the difficulty of separating those minerals.

The use of 2% sea water, Figure 4, decreases generally the zeta potentials and shifts the zero point of charge of magnetite to a lower value. All minerals do, in addition, get a reversal of zeta potential between pH 10 and 11. Reversal of charge for iron-bearing pyroxenes in the presence of divalent cations has already been reported by Fuerstenau et al (7). Higher sea water concentrations may move the charge reversal to lower pH's. Quartz is still the only mineral that has a distinctively lower zeta potential than the other minerals. Apparently a difference of 5-10 mV in zeta potential is enough to make selective flotation possible.

The reason why diamine might float amphiboles around pH 7 and 8 is not clear. We might be dealing with a more complicated adsorption mechanism which can work on slight differences in zeta potential. The minerals present also display their minimum zeta potentials in the pH range 5-8 which could give a maximum dispersion.

CONCLUSION

Magnetic separator taconite concentrates, where the predominant impurity is quartz, can be upgraded to more than 70% Fe by flotation, at a good recovery. Straight-chain amines should be used as collectors between pH 9 and 10 and starch as depressant.

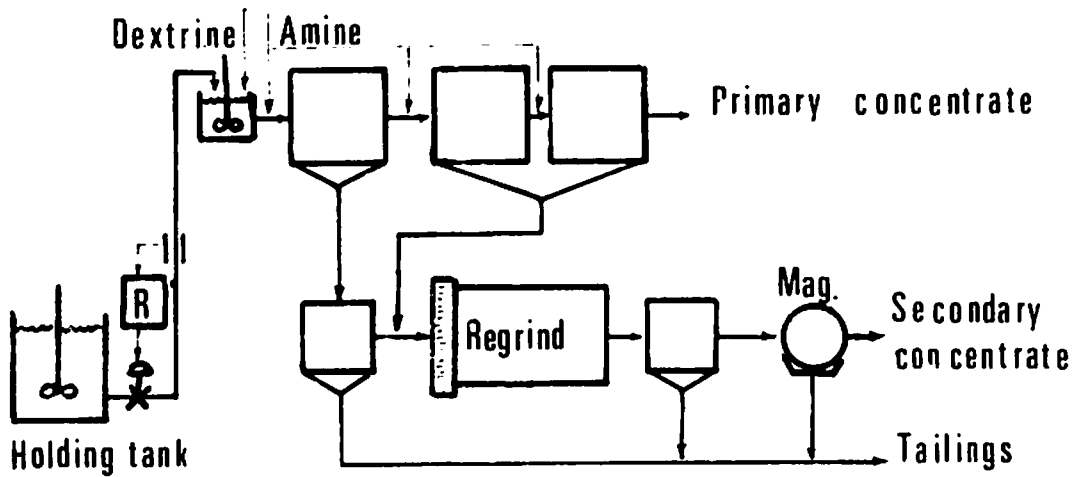
Magnetic products, where important gangue minerals are hornblende and cummingtonite, do not respond well to the above treatment, especially in the coarser size range. Diamines at pH 8 make it possible to obtain good grades under those conditions, but at the expense of total iron recovery in a straight float. Much of the iron loss can be recovered by middlings treatment such as refloatation or magnetic separation, but this product will be very fine and have a grade close to flotation feed.

The use of sea water and pulp of low temperature will generally lead to lower-grade concentrates compared to what can be obtained in fresh water at room temperature. It has, however, been noted that amphiboles float best at low temperatures.

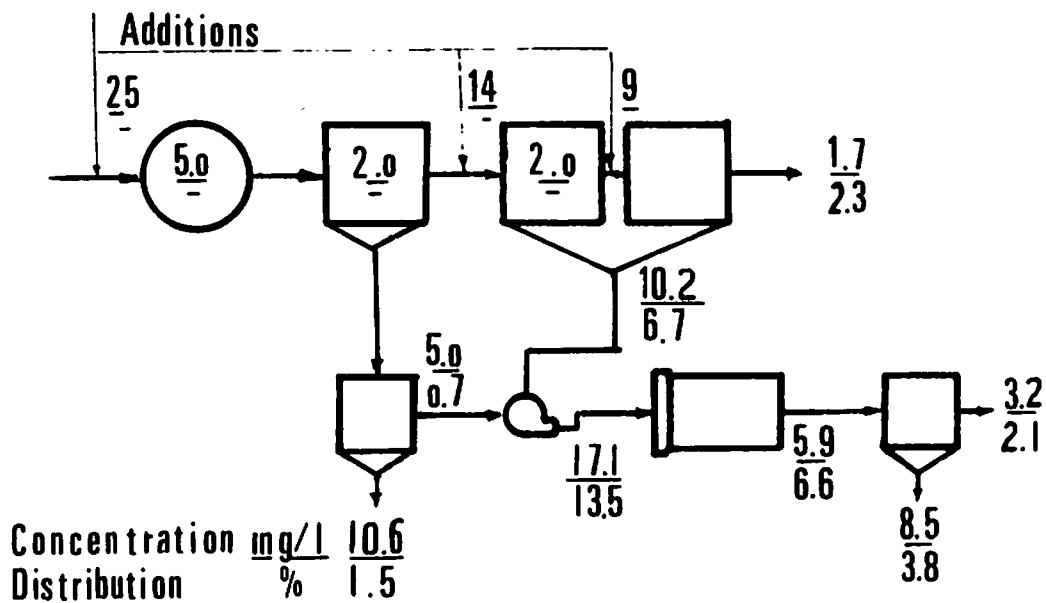
Comparative zeta potential measurements of magnetite, quartz, hornblende and cummingtonite have shown that quartz is the only mineral that has a definitely lower zeta potential than the others. Quartz should therefore be relatively easy to float, while the separation of magnetite, hornblende and cummingtonite should give problems like the ones encountered.

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PILOT FLOWSHEET SILICATE FLOTATION
 Figure 1 Sydvaranger ore



DIAMINE CONCENTRATIONS IN THE WATER PHASE
 Figure 2 Neutral pulp

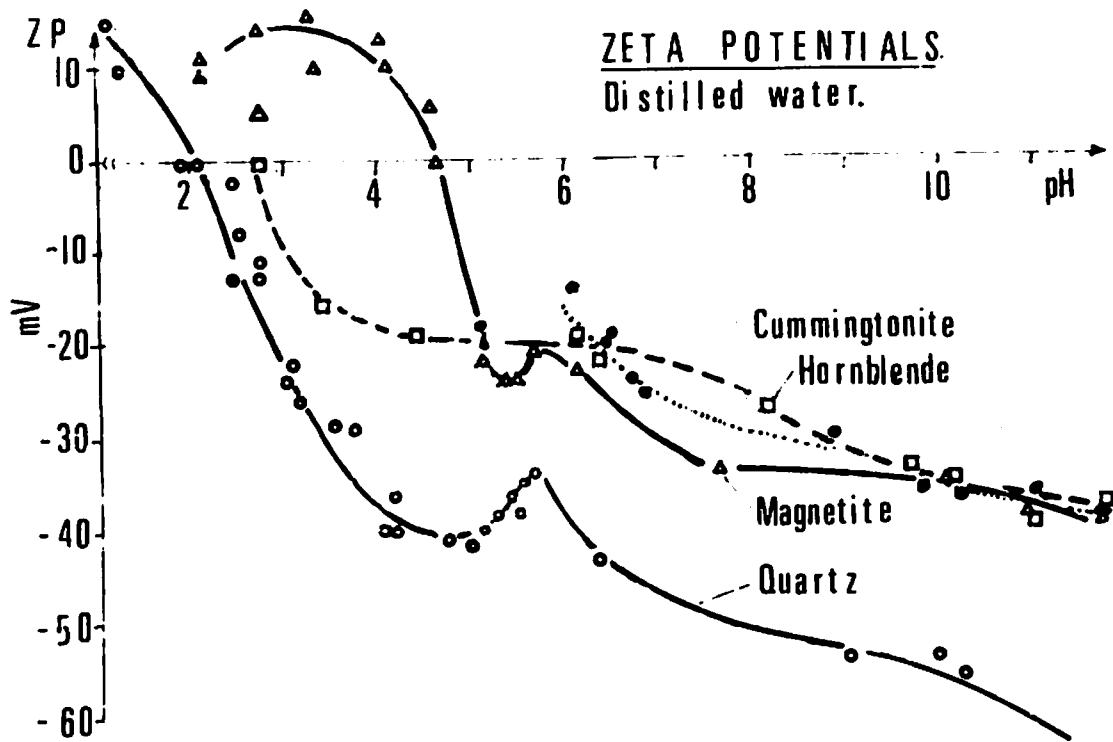


Figure 3

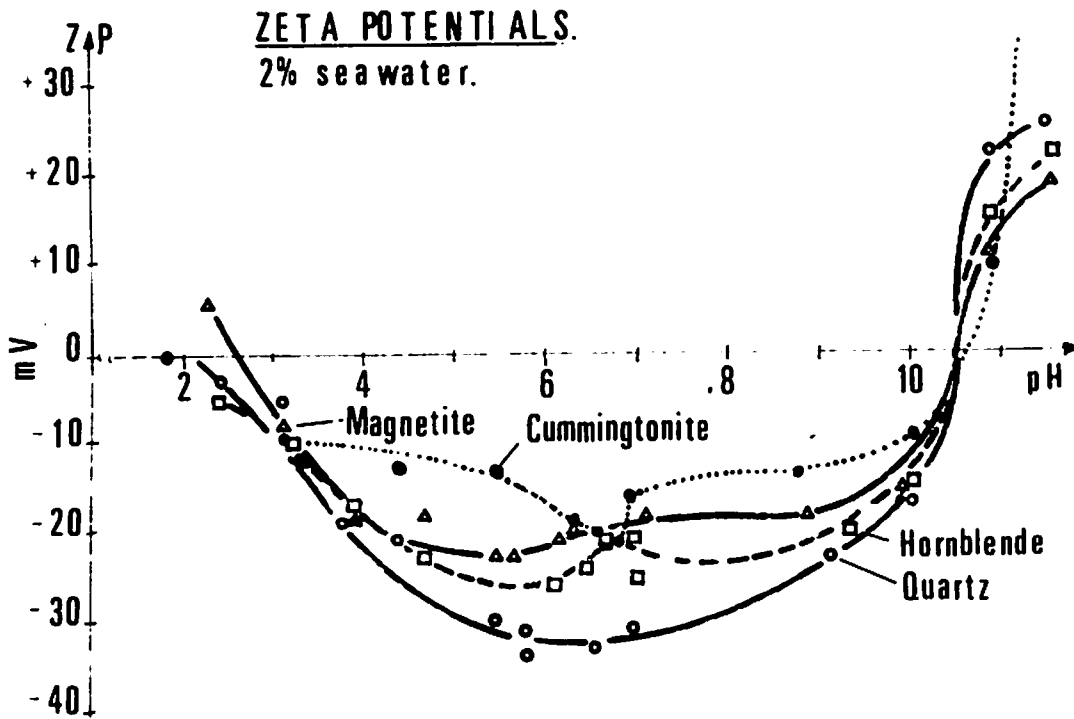


Figure 4